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Bulletin No. 6

1911

MEMORANDUM ON THE OIL VALUE OF SOME SANDAL WOODS FROM MADRAS

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FM-194: Memorandum On The Oil Value Of Some Sandal Woods From Madras

BY

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1911

MEMORANDUM ON THE OIL VALUE OF SOME SANDAL WOODS FROM MADRAS.

By PURAN SINGH, F.C.S.,
Forest Chemist

IT is generally believed that the wood of the Sandal trees growing in dry rocky mountainous soil is richer in oil than that of the trees found in the more fertile soils of the plains. Considering this point to be of some commercial importance, the Forest Chemist, in consultation with Mr. Lodge, Conservator of Forests, Madras, proposed to determine the variation in the oil values of trees growing in different localities on different kinds of soil. Mr. Lodge kindly supplied 15 different specimens of Sandal wood from trees growing on different kinds of soil for examination, and this memorandum embodies the results obtained which, it will be seen, go to confirm the general belief stated above.

It was not intended to work out the maximum and minimum percentage of oil in Sandal wood generally, as this is already known to range between 3 and 5 per cent., according to Gildmeister and Hoffman [1] and 1·5 and 6 per cent. according to Parry. [2] Though the statement of the maximum and the minimum oil values requires the examination of many more specimens of Sandal wood than it was found possible to examine, yet the 15 samples of Sandal wood mentioned above appear to cover a sufficiently wide range of variation in quality, while the percentage of oil in them ranges between 0·85 and 5 per cent. These figures approximate to the figures of Parry.

General remarks on the Distillation of Sandal Wood Oil.

Sandal wood oil is obtained by distilling perfectly comminuted wood of *Santalum album* Linn. (Family Santalaceæ), by superheated steam in

[1] *Vide* Volatile Oils by Gildmeister and Hoffman, Edit., 1900, p. 340.
[2] *Vide* The Chemistry of Essential Oils by E. J. Parry, Edit., 1899, p. 181.

suitable forms of steam stills, of which some special designs are the patents of Messrs. Schimmel & Co., Leipzig.

Owing to the more up-to-date processes of distillation, the oil distilled in Europe is better in quality than that distilled in the primitive method obtaining in India. The former is light coloured, and has a pleasant odour, while the latter is more or less contaminated with empyreumatic products towards the later stages of crude distillation, and is consequently of a higher specific gravity and a darker colour.

Bidie[1] describes the native process as follows :—

"The body of the still is a large globular clay pot, with a circular mouth, and is about 2½ feet deep by about 6 feet circumference at the bilge. No capital is used, but the mouth of the still, when used, is closed with a clay lid, having a small hole in its centre, through which a bent copper tube about 5½ feet long is passed for the escape of the vapour. The lower end of the tube is conveyed inside a copper receiver, placed in a large porous vessel containing cold water. When preparing the Sandal wood for distillation the white or sap-wood is rejected, and the heart-wood is cut into small chips, of which about 2 mds. or 50 lbs. are put into the still. As much water is then added as will just cover the chips, and the distillation is carried on slowly for 10 days and nights, by which time the whole of the oil is extracted. As the water from time to time gets low in the still, fresh supplies are added from the heated contents of the refrigerator."

Troup in his Manual of Forest Utilization, p. 224, gives almost the same description of the Sandal wood distillation in India and says that sometimes the distillation is carried on for 21 days.

The yield of the oil as noted above is very variable; in Europe they get about 3 to 6 per cent. and in India it is said to average 2·5 per cent. It is possible that by using chips of the wood, a larger yield is not obtained by the Madras distillers. The percentage of the oil, however, as obtained by the Kanauj distillers of the United Provinces, is stated to be never below 5 and 6 per cent. The distillation as done in Kanauj is nearly of the same description as that described by Bidie and Troup, with this difference that the Kanauj distillers reduce the Sandal wood to a fine powder by filing, and soak the powder in water for 48 hours before placing it in a copper still tinned inside. They change their

[1] Holmes, Pharm. Journ., III, 16, p. 819.

receivers directly they are full, return the watery layer of the distillate at certain intervals to the still, and continue the distillation for about a week, day and night, maintaining a uniform heat and observing the utmost cleanliness. According to the Kanauj perfumers a Sandal wood yielding below 5—6 per cent. of oil is inferior wood of comparatively low value. The writer has reliable information that the maximum yield of 6 per cent. as given by Parry is obtained in practice from superior kinds of Sandal wood by the Kanauj distillers with their apparatus, however crude it may be. This information is also confirmed by Sir George Watt, who in the "Commercial Products of India," page 978, says that the yield in Kanauj is reported to be about $2\frac{1}{4}$ to 3 seers of oil to 1 maund ($=82\frac{2}{7}$ lbs.) of wood. The oil distilled at Kanauj is quite free from objectionable odour.

The writer's information also confirms the statement made by Sir George Watt that the annual outturn at Kanauj is at the lowest about 8,000 lbs. of the oil. It is said, however, that for want of fuel and owing to the increasing price of good Sandal wood this industry is fast declining. It may, therefore, be appropriately pointed out here that the establishment of an up-to-date factory in perfumery centres like Kanauj, where large quantities of Sandal oil are consumed every month in the manufacture of Indian *attars* and perfumes might reasonably prove a profitable concern. The introduction of the modern methods of distillation would considerably reduce the cost of production of the oil. The plant required has to be of special design consisting of the following :—

(1) A rasping machine, with which to comminute the Sandal wood into fine filings.

(2) An efficient form of steam still complete with tubular condenser, florentine flasks, etc.

(3) A small copper still for re-distillation of the oil.

(4) A boiler capable of generating superheated steam.

The total cost of this distillery, the writer estimates at about R40,000. The best Sandal wood yielding about 6 per cent. of oil is obtainable at R35 a maund. One maund would, therefore, yield about $4\frac{4}{5}$ lbs. of oil worth R43, taking the London price of Sandal wood oil at £0-12-0 a lb. Besides supplying the oil to the perfumery manufacturers in India, there is every possibility of an export trade being developed in this article. (See Appendix.)

The writer wishes to point out here that ordinary steam at 30 lbs. pressure as commonly used in the distillation of other essential oils cannot be recommended for distillation of Sandal wood, as a fairly large quantity of the oil during the process of distillation is apt to be lost in the copious flow of condensed water in a semi-emulsified form, and even 24 hours continuous distillation with steam at that pressure does not bring out more than one-third of the total quantity of the available oil. This oil has an abnormal tendency to form an emulsion with water, and for this reason, unless a really efficient and up-to-date plant is put up, it will be an unwise step to try to replace the so-called primitive method by steam distillation, as practised in the distillation of the volatile oils generally.

The Chemistry of the Sandal Wood Oil.

The Sandal wood oil is rather viscid, pale to light yellow liquid of characteristic aromatic odour. Its specific gravity ranges between ·975 and ·980. Parry mentions his having met with some genuine samples with specific gravity ranging between ·973 and ·981. The optical rotation of the Sandal wood oil is a very characteristic test of its purity. It seldom varies outside the limits of —15° to —20°. With 5 parts of 70 per cent. alcohol it yields a clear solution which is not rendered turbid by any further addition of alcohol. It contains small quantities of saponifiable matter requiring for saponification from ·7 to 1·5 per cent. of caustic potash. After acetylation, however, the percentage of caustic potash required rises to 20—21 per cent., thus showing that this oil mainly consists of unsaponifiable alcohols.

The acetylation process first applied by Parry in the examination of Sandal wood oil is an important method for the chemical examination of this oil. It is carried out as follows:—About 20 grams of the oil are gently boiled for 1½ hours over a water bath under a reflux condenser with an equal volume of acetic acid anhydride and a small quantity of fused sodium acetate. The product consists of acetic ester of santalol (C_{15} H_{26} O) having the formula C_{15} H_{25} O. $COCH_3$. The santalol ester is diluted with water and is kept on the water bath for a few hours to decompose the excess of acetic anhydride present. The product thus treated is then thoroughly washed with water and soda solution till the washed water is perfectly neutral in its reaction. It is then dried over anhydrous sodium sulphate, and 2—5 grams of this dried oil are taken for the

determination of its saponification value. The amount of santalol is then calculated by the following formula :—$X = \frac{N \times M}{10\ (W - 0{\cdot}42\ N)}$ where X is the percentage of alcohol in the original oil, M is the molecular weight, N is the number of cc. of $\frac{N}{I}$ alkali used for saponifying the acetylated oil and W is the weight of the acetylated oil taken for saponification.

The determination of the santalol contents is the best means of examining the purity of Sandal wood oil. Good oils mostly show 93—98 per cent. santalol content and never contain less than 90 per cent. Parry gives the following results of his examination of the several samples of Sandal wood oil :—

Sample.	Specific gravity.	Saponification figure (per cent. KOH).	Boiling point 11 mm. pressure.	Percentage of santalol calculated for $C_{15}\ H_{26}O$.
1	·979	·13	168° to 169°	98·8
2	·980	·63	...	100·3
3	·979	·80	168° to 170°	99·3
4	·979	·35	...	100·1

The Sandal wood oil according to Chapoteaut[1] consists of an aldehyde of the formula $C_{15}\ H_{24}\ O$ boiling at 300°C. and a corresponding alcohol of the formula $C_{15}\ H_{26}\ O$ boiling at 310°C. These results are not generally accepted as correct because the oil used by this author was of a very low specific gravity, *viz.*, 0·945, and hence it is supposed it was an adulterated specimen.

Von Soden and Muller[2] found in the non-alcoholic portion of the oil a sesquiterpene of sp. gr. 0·898, d= α—21° and having a boiling point of 261°—262°C. It could not be indentified with any of the known sesquiterpenes.

According to Guerbet[3] who is also reported to have worked on an impure specimen, the Sandal wood oil has the following constituents ;—

1. Two sesquiterpenes, α—santalene, b.p. 252-252·5°C., sp. gr.

[1] Bull. Soc. Chim. II, 37, p. 303.
[2] Pharm. Zeiting 44, p. 258.
[3] Compt. rind. 130, p. 417, Journ. de Pharm. et. Chim. VI 9, p. 224.

0·9134 at 16°C., $\alpha d = -13·98°$, β—santalene, b.p. 261-262°C, sp. gr. 0·9139 at 0°, $\alpha d = -28·55°$.

2. A mixture of two alcohols of the composition $C_{15} H_{26} O$ of different rotatory power.
3. An aldehyde $C_{15} H_{24} O$, santalol b.p. 180° at 40 mm. having a strong pepper like odour.
4. An acid $C_{15} H_{24} O$, santalic acid boiling at 210-212° at 20mm., being a colourless, viscid liquid.
5. An acid $C_{10} H_{14} O_2$, teresantalic acid m.p. 157°.
6. Traces of strongly aromatic substances to which is due, to a large extent, the odour of the oil.

It will thus be seen that the subject of the chemical composition of this interesting oil is still an open one, and it is further being investigated by Parry.

Methods of Estimation of the Volatile Oil in Sandal Wood.

The estimation of the volatile oil in Sandal wood, according to the writer's practice, can be carried out by three different methods which give fairly concordant results :—

I. About 500 grams of the Sandal wood filings are steam distilled. The distillate consists of a milky emulsion of water and oil with particles of the oil floating on its surface. The process of distillation lasts about a week and it is to be considered complete when the distillate runs out perfectly clear with no particles of oil suspended in or floating on it. The whole of the day's distillate is collected in a bottle, shaken with chloroform and left overnight. The watery layer which during the night separates out clearly from the under layer of chloroform, is siphoned off and the second day's distillate is taken again and shaken with the same chloroform. This operation is repeated every day till the total quantity of the oil distilled in seven days is taken up by the chloroform. The chloroform after being filtered into a tared flask is distilled off and the residual oil is freed from the last traces of chloroform by blowing a gentle current of air into it for a few minutes and weighed. This process is very lengthy,

but it gives reliable results because of the large quantity of material used. It could not be adopted for general use in the laboratory, but it is the only one which gives results to which those obtainable on a commercial scale can be made to approximate. Any process adopted in the laboratory must be referred to it for comparison.

II. About 10—15 grams of Sandal wood filings are extracted with ether in a Soxhlet's extraction apparatus for about 12—14 hours necessary to complete the extraction. Ether extract thus obtained, after the whole of ether is distilled off, is subjected to steam distillation as in No. I. The steam distillation of the ether extract takes only about 4-5 hours. For the estimation of the oil, the total distillate is treated as in No. I. This process is generally adopted for the estimation of volatile oils in woods and barks in the laboratory, but in the case of sandal wood oil it uniformly gave low figures. It is just possible that the ether extraction done as above is insufficient and traces of oil are left in the wood powder. The results obtained by this process are in many cases, however, in fair agreement with those obtained with process No. I.

III. About 10—15 grams of the Sandal wood filings are extracted with alcohol three times successively over water bath under reflux condenser, using small quantities of alcohol at a time. To avoid bumping, some glass beads should be added. It was seen that instead of extracting hot as above, the same results could be obtained by macerating Sandal wood filings in a sufficient quantity of alcohol for a week. The alcoholic extract after the whole of alcohol is distilled off is treated as in Nos. I and II. In addition to the distillation of alcoholic extract, the residual Sandal wood powder should also be distilled to collect any traces of the oil that may still be in the wood. For the estimation of the oil the total distillate is treated as mentioned above. This process was found to be very speedy and accurate. The figures obtained by it were in close agreement with the steam distillation figures. One estimation takes about 5-6 hours. The writer is of opinion that this method is best suited for the determination of oil in sandal wood.

Description of the Specimens examined.

The description of 15 specimens received from Mr. Lodge for examination is tabulated below:—

Serial number.	Description of the specimen.	Name of the locality from where obtained.	Description of the soil in which the tree was growing.
1	Stem .	North Coimbatore .	Not mentioned.
2	" .	South Canara District	Deep, porous laterite soil. Elevation between 400 and 500 feet.
3	" .	Do. .	Hard, laterite soil. Elevation about 400 feet.
4	Roots* .	Nilgiris . . .	Extracted from a tree on the bank of a stream in a shady spot. Elevation about 3,000 feet, soil alluvial and deep. Associates bamboos.
5	Stem* .	Do. . . .	Ditto ditto.
6	Roots* .	Do. . .	Extracted from a tree 6 miles distant from the tree from which samples 4 and 5 were extracted. Elevation 3,000 feet. Soil poor gravelly. Associates *Acacia* and *Zizyphus*.
7	Stem* .	Do. . . .	Ditto ditto.
8	" .	North Coimbatore .	Soil hard and rocky, but fertile for cultivation. Elevation 2,700 feet.
9	" .	Do. .	Soil is hard and rocky. Elevation 3,800 feet.
10	" .	Do. .	Soil loose conglomerate with black cotton soil. Elevation 3,160 feet.
11	" .	Do. .	Soil clay. Elevation 3,020 feet.
12	" .	Do. .	Soil gravelly. Elevation 3,300 feet.
13	" .	Do. .	Soil is sandy loam resting on a sub-soil of decayed granite.
14	" .	Do. .	Soil consists of rich loam for about 6 inches deep and the sub-soil of disintegrated rock. Elevation 2,500 feet.
15	" .	Do. .	The soil is red loam mixed with large quantity of sand. Elevation 2,800 feet.

* **Note.**—The Ranger states that these two pairs of specimens are types of the wood grown on good and bad soils in his Range and that the specimen Nos. 4 and 5 are more highly scented.

It will be seen that excepting specimens 4 and 6 which were roots of trees growing in different kinds of soils, all the other specimens were pieces taken from the stem, some of the pieces being rather light and others heavy. The following table shows the percentage of moisture and oil in them :—

Specimen number.	Moisture.	Oil by alcohol and steam.	Oil by ether and steam.	Oil by steam alone.	REMARKS.
	Per cent.	Per cent.	Per cent.	Per cent.	
1	13·32	3·94	...	...	
2	15·37	0·78	0·39	...	Mostly sap-wood.
3	14·63	1·83	0·62	...	Poor specimen.
4	14·47	2·28	1·90	...	
5	15·01	0·48	0·30	...	Mostly sap-wood.
6	13·61	3·07	2·50	...	
7	14·77	0·63	...	...	Mostly sap-wood.
8	13·70	3·26	3·17	...	
9	13·16	5·02	4·65	...	
10	14·42	3·64	3·03	...	
11	14·19	4·24	...	...	
12	13·93	1·96	...	...	Poor specimen.
13	13·50	3·97	...	4·07	
14	14·69	4·44	4·25	...	
15	16·20	3·75	3·60	4·33	

Of these specimens it will be seen that Nos. 2, 3, 5, 7, 12 give very low values. This is due to the fact that they were mostly sap-woods, having very little heart-wood. Of the specimens 4 and 6, it will be seen that 6 growing in soil more rocky than No. 4 is richer in oil than the latter. Of the specimens 8, 9, 10, 11, 13, 14 and 15, Nos. 9, 13, 14 and 15 are richer in oil than 8, 10, 11. A reference to the table of the

description of the samples shows that Nos. 8, 10 and 11 were taken from the trees growing on comparatively fertile soils, while Nos. 9, 13, 14 and 15 were taken from the trees growing on poor, rocky and gravelly soils.

This conclusion seems to be in accord with the opinion expressed by Gildmeister and Hoffman (*vide* "Volatile Oils" by these authors, p. 339) that the wood of the tree growing in dry, rocky, mountainous soil is harder and richer in oil than that of trees growing in fertile soils.

In conclusion the constants of the Sandal wood oil as determined in the Laboratory of the Forest Research Institute may appropriately be given here. The oil used for the determination of constants was the average sample made up by mixing the different quantities of oil obtained in the estimation distillations :—

Specific gravity at 26°C.	·9765
Optical rotation	—15·6°—16°
Saponification number before acetylation	9·72
Do. after acetylation	21·13
Santalol content	99·4 per cent.

APPENDIX.

The distillation of Sandal wood oil is, in the writer's opinion, full of promise. A large quantity of the oil is consumed by the native perfumers. The Kanauj perfumers alone probably consume about 10,000—15,000 lbs. a year. It may be, therefore, of some interest to the enterprising capitalist to form an idea of the profitable nature of the industry.

The following is a rough estimate of the working of a sandal wood distillery. The factory may be best started in Bombay, Mysore, Madras or Kanauj :—

Capital outlay for Machinery and Buildings, Rs. 40,000.

Profit and Loss account for one year's working.

Dr.	R	*Cr.*	R
Cost of 7,320 mds. of good sandal wood (yielding 5 per cent. oil) at R30 a maund (= $82\frac{2}{7}$ lbs.)	2,19,600	Annual output of oil 30,000 lbs.	
Cost of establishment, labour and fuel	15,000		
Depreciation at 10 per cent.	4,000	Sale proceeds of oil at R9 a lb.	2,70,000
Renewals and repairs	2,000		
Contingencies	3,000		
Total	2,43,600		
Nett profit	26,400		
GRAND TOTAL	2,70,000	TOTAL	2,70,000

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